

# Enrichment of Pasteurized Whole Milk with Iron

## Abstract

Organoleptic evaluation of iron-enriched whole milks showed that ferric iron compounds uniformly resulted in lipolytic rancidity when milk was pasteurized at minimum to moderate temperatures (below about 79 C). This off-flavor was reduced acceptably, or completely eliminated simply by pasteurizing at 81 C. Data consistently showed that milk lipase is made more heat resistant by ferric forms of iron.

Ferrous compounds normally caused definite oxidized flavor when added to raw whole milk before pasteurization. This off-flavor was markedly reduced by de-aerating the milk before adding the iron. Ferrous compounds did not cause rancidity.

Ferric ammonium citrate and ferrous sulfate were evaluated more extensively than other compounds. Addition of the citrate salt followed by pasteurization at 81 C is the more feasible method, since it would be difficult to add the ferrous salt after deodorization and before pasteurization.

## Introduction

Iron deficient diets are reported for several million Americans in a summary of a food consumption survey made in 1965 (2). Children below 3 years of age were receiving only about 50% of recommended dietary allowances (RDA). Girls and women from age 9 to 54 years were receiving only 65 to 70% of RDA's for both iron and calcium. The calcium intake of children (both sexes) below 8 years of age was sufficient or above recommended amounts.

These results are especially significant for the role milk has in our diets. About three-fourths of the Ca in diets of Americans is supplied by milk. The Ca deficiencies of the groups indicated could be corrected by increased milk consumption. Correction of the iron deficiencies, however, is a different problem. Milk and milk products are low in iron. They are, however,

available everywhere and are consumed in substantial amounts by most people. Thus, they are appropriate foods for enrichment with iron.

Enrichment of milk with iron is not new. Most states have standards for adding iron to milk (whole milk, low-fat milk and skimmilk) along with iodine and several vitamins (1). The amount of these products marketed is not known. Also, there is little published information concerning problems with flavor and processing conditions which affect the quality of iron-enriched milk products.

The dairy industry has for years avoided the contamination of its products with iron. This is because iron catalyzes the development of the oxidized flavor, although iron is not as effective as copper. Nevertheless, it is implicated.

Scanlon and Shipe (5) reported on the susceptibility of multi-vitamin mineral (MVM) milk to oxidation. They concluded that ferrous iron is responsible for the catalytic oxidation of milk when iron compounds are used for mineral fortification of whole milks. Reisfeld et al. (4) reported that the addition of ferric ammonium citrate to milk protected lipase against heat inactivation at a normal pasteurization temperature of 73.3 C for 16 sec.

This study was undertaken to evaluate pasteurized whole milks enriched with several different iron compounds and to develop optimum processing conditions necessary to minimize off-flavors resulting from the iron.

## Experimental Procedures

In early trials, iron-enriched samples were prepared from deodorized milk to remove feed flavors, which to some extent masked iron-induced off-flavors. Deodorization consisted of heating raw whole milk to 72 C for 16 sec and "flashing" into a vacuum pan without heat. A portion of the deodorized milk was reserved for a control. Other lots were fortified with different iron compounds. In most trials the additions of iron were made immediately before pasteurization which followed deodorization but in a few cases, for comparative purposes, the iron was added after pasteurization. Pasteurizing temperatures were varied and are specified

Received for publication April 19, 1971.

<sup>1</sup> Southeastern Marketing and Nutrition Research Division, ARS, USDA, Athens, Georgia 30604.

in the Discussion section; pasteurizing holding time was 16 sec in all cases.

Since preliminary deodorization as indicated is not a feasible arrangement for many commercial plants, additional samples were prepared from nondeodorized milk. In these trials the iron was added to raw milk followed by pasteurization and homogenization. Some of the samples pasteurized at minimal to moderate temperatures exhibited a rancid (lipolytic) flavor; others an oxidized flavor. Since rancidity is caused by lipase, this phase included an evaluation of milks pasteurized at 72 to 82 C. The effect of the type of iron compound and of processing with and without deodorization on oxidized flavors was also included in this series of experiments.

The iron compounds were dissolved or suspended in water prior to mixing with the milk. Iron concentrations in the water were usually 10 mg per milliliter of solution.

Organoleptic evaluations of the processed milks were made by either of two methods: a) a trained panel of 10 members using a modified ADSA scoring system (3), and b) a flavor intensity rating method. The latter method determined the effect of type of iron compound and of processing procedures on the intensity of rancid and oxidized flavors. The judges were instructed to use the rating scale: 0 = no off-flavor, 1 = questionable, 2 = slight, 3 = distinct, and 4 = strong. A panel of 6 judges was used for this method.

#### Results and Discussion

The results of taste panel evaluations of samples from deodorized milk and containing various iron compounds are in Table 1. Most of the iron-enriched samples (except ferrous fumarate) had flavor scores near those of the controls. The sample containing ferric choline citrate had an unacceptable flavor after one day but improved after one and two weeks. Several other samples showed improvement with age. The improvement may be real and is associated with a decreased frequency of oxidized flavor. For example, 65% of samples scoring less than 35.5 had oxidized flavor, whereas only 13% of those scoring above 36.5 were oxidized. The ferrous compounds showed a greater tendency to develop an oxidized flavor than the ferric compounds, although at 10 mg of Fe per quart and when processed as indicated, most of the compounds used appear to be acceptable additives for enriching milk.

Many plants pasteurize whole milk at high temperatures, or with vacuum deodorization.

TABLE 1. Effect of addition of iron compounds<sup>a</sup> on flavor of pasteurized whole milk.<sup>b</sup>

Compound	Flavor score <sup>c</sup>		
	1 Day	1 Week	2 Weeks
Ferric ammonium citrate	36.1	36.3	36.6
Ferric choline citrate	34.3	36.1	36.6
Ferric glycerophosphate	37.2	36.5	36.5
Ferrous sulfate	35.9	36.4	36.5
Ferrous ammonium sulphate	36.4	36.7	36.7
Ferrous fumarate	34.3	34.5	35.3
Ferrous gluconate	35.6	36.3	36.2
Control	37.2	36.5	36.7

<sup>a</sup> Added iron = 10 mg/qt.

<sup>b</sup> Milks were "deodorized" by heating to 72 C for 16 sec., iron added and pasteurized at 74.5 C for 16 sec.

<sup>c</sup> Averages of two trials; nine judges per panel. Scoring range = 31 to 40; score 35 considered acceptable.

The effect of pasteurization temperature on the flavor of an iron-enriched milk is shown in Table 2. Also, the effect of adding iron after pasteurization on flavor is shown. It is evident that pasteurization at 84 C of the iron-enriched sample resulted in an unacceptable flavor, whereas the addition of iron after pasteurization at 84 C had no effect on flavor when compared to the control. The predominate criticism of the high-heat, iron-enriched sample was oxidized. The flavor of some of the samples was described as being atypically oxidized.

As noted earlier some of the iron-enriched samples developed a rancid flavor. To determine whether only certain compounds caused this off-flavor and to what extent, a series of raw milk samples fortified with several different iron compounds was pasteurized at near minimum temperatures. The first group of samples in this study were fortified with 40 mg of iron per quart. For this series the taste panel judges were instructed to rate the intensity of rancid, oxidized, and feed flavors. They also had the option of listing the intensity of other flavors if observed. Flavor analyses are in Table 3. The differences between the

TABLE 4. Effect of pasteurization temperature on the intensity of rancid and oxidized flavors in iron enriched<sup>a</sup> whole milks.

		Flavor intensity					
		Rancid			Oxidized		
		Days of storage					
Iron compound	Pasteurized at:	1	7	14	1	7	14
	(C)						
Ferrie ammonium citrate	72.2	xxx	xxxx				
	74.5	x	xxxx				
	76.7	x	xxx	xxx			x
	79.4		x	xx			x
	81.1		x	x			x
	82.2			x			
Ferrous sulfate	72.2				xxxx	xx	
	74.5				xxx	xx	
	76.7	x			xxx	xxx	

<sup>a</sup> Added iron = 10 mg per quart.<sup>b</sup> Flavor intensities were by a 6-judge panel using the following scale: Blank = none, x = questionable, xx = slight, xxx = distinct, xxxx = strong.

iron increased the heat resistance of milk lipase. A temperature of about 81 C is sufficient to reduce lipolysis acceptably. The inactivation temperature (with a 16-sec holding time), no doubt, will depend on the amount of iron added, the type of iron compound, and possible prior treatment of milk such as agitation. Some studies with variable homogenization pressures indicate that the higher the homogenization

pressure, the greater the tendency to develop a rancid flavor. This effect of course, applies only to milks containing residual lipase. Homogenization up to 281.2 kg/cm<sup>2</sup> after pasteurization was employed.

The degree of rancidity, if present in freshly pasteurized samples, increased with storage time, whereas the oxidized flavor in the ferrous-fortified samples decreased during storage.

TABLE 5. Effect of deaeration on intensity of oxidized flavor in iron-enriched whole milk.<sup>a</sup>

Sample treatment			Flavor intensity <sup>b</sup>					
			Oxidized		Rancid		Feed	
			Days of storage					
De-odorized <sup>c</sup>	De-aerated <sup>d</sup>	Fe added	1	7	1	7	1	7
(mg/qt)								
No	No	10	3.0	2.2	0	0	0	0
Yes	No	10	0.5	0.3	0	0	0	0
No	Yes	10	0.8	0.5	0	1.1	0	0
No	No	20	3.0	3.0	0	0	0	0
Yes	No	20	1.7	0.5	0	0	0	0
No	Yes	20	1.0	2.0	0	0	0	0
No	No	Control	0	0.3	0	0.6	1.7	1.3
Yes	No	Control	0	0.2	0	0	0.7	0.8

<sup>a</sup> Iron source — FeSO<sub>4</sub>; milk pasteurized at 74.5 C for 16 sec after addition of iron.<sup>b</sup> See footnote Table 3.<sup>c</sup> Deodorized before adding iron (See Experimental Procedures).<sup>d</sup> Deaerated by bubbling nitrogen through raw milk before adding iron.

TABLE 2. Effect of temperature of pasteurization on flavor of iron enriched whole milk.

Iron compound <sup>a</sup>	Pasteurized at:	Fe added before or after past.	Flavor score <sup>b</sup>	
			1 Day	14 Days
	(C)			
Ferric ammonium citrate	74.5	Before	36.4	36.8
	84	Before	34.4	34.5
	74.5	After	36.5	36.8
	84	After	36.2	37.2
Controls	74.5	.....	37.2	37.0
	84	.....	36.3	37.0

<sup>a</sup> Added iron = 10 mg/quart.<sup>b</sup> See footnotes to Table 1.

ferric and ferrous compounds on the resulting off-flavors in these samples are striking: ferric compounds caused a rancid flavor, whereas the ferrous compounds resulted in an oxidized flavor with no rancidity. Rancidity and oxidized flavors in the controls were insignificant.

Another series of samples were enriched with either ferric ammonium citrate or ferrous sul-

fate at 10 mg of iron per quart and pasteurized at 72.2 to 82.2 C. Flavor ratings after 1 and 7 days of storage at 4.4 C are in Table 4. Again the differences in flavor between ferric and ferrous enriched milk samples are marked. The intensity of the rancid flavor in the ferric-fortified samples decreased with increasing temperature. It may be concluded that added ferric

TABLE 3. Effect of added iron<sup>a</sup> on rancid and oxidized flavors in pasteurized whole milk.

Iron compound	Pasteurized at:	Flavor intensity <sup>b</sup>		pH
		Rancid	Oxidized	
	(C)			
Ferric ammonium citrate	71.0	4.0	0	6.45
	72.2	3.0	0.3	6.55
	73.3	3.2	0.3	6.51
Ferric glycono-phosphate	72.2	3.5	0.8	
	73.3	1.5	1.2	
Ferric citrate	72.2	1.5	1.3	
	73.3	1.7	2.2	
Ferrous gluconate	71.0	0	3.5	6.64
	72.2	0.2	3.3	6.66
	73.3	0.2	3.5	6.64
Ferrous ammonium sulfate	72.2	0	2.8	
	73.3	0.2	3.3	
Ferrous sulfate	72.2	0	3.0	
	73.3	0	3.8	
Control	71.0	2.0	0	6.65
	72.2	0.8	0	6.66
	73.3	0.3	0	6.69

<sup>a</sup> Added iron = 40 mg per quart.

<sup>b</sup> Taste tests were made one day after processing. Flavor ratings are averages of 6 judges. Judges were instructed to use the following rating scale: 0 = none, 1 = questionable, 2 = slight, 3 = distinct, 4 = strong.

Data in Table 1 show that some of the ferrous-fortified samples were acceptable; only slight oxidized criticisms being indicated in most cases. However, definite to strong oxidized flavors were observed in similarly fortified samples which were not pre-deodorized (Tables 3 and 4). The beneficial effects of oxygen removal prior to ferrous iron additions are evident. A more direct comparison of the effects of deaeration on oxidized flavors in ferrous iron-fortified whole milks is shown in Table 5.

#### Conclusions and Recommendations

The addition of ferric iron salts to raw whole milk increases the heat resistance of lipase, resulting in rancid flavor in milks pasteurized at below 79 C. Rancidity can be overcome simply by pasteurizing at 81 C which inactivates lipase.

The ferrous salts caused an oxidized flavor. This problem can be overcome by deaerating

the milk before adding iron. Pasteurization should then follow the addition of the iron.

#### References

- (1) Federal and state standards for the composition of milk products. Agriculture Handbook 51, Consumer and Marketing Service, USDA, Washington, D.C. Revised July 1968.
- (2) Food intake and nutritive value of diets of men, women and children in the United States, Spring 1965. A Preliminary Report, ARS 62-18. March 1969.
- (3) Liming, Naomi E. 1966. Consistency of a trained taste panel. *J. Dairy Sci.*, 49: 628.
- (4) Reisfeld, R. A., W. J. Harper, and I. A. Gould. 1955. The effect of mineral fortification on lipase activity in pasteurized milk. *J. Dairy Sci.*, 38: 595.
- (5) Scanlan, R. A., and W. F. Shipe. 1962. Factors affecting the susceptibility of multivitamin mineral milk to oxidation. *J. Dairy Sci.*, 45: 1650.